

State of New York )

SS:

County of New York

### AFFIDAVIT

On this day, Robert C. Ferber personally appeared before me and after being duly sworn, deposes and states:

- 1. That he is well qualified as a translator of German to English and is employed as such by Kenyon & Kenyon (One Broadway, New York, New York 10004);
- 2. That he has carefully reviewed the attached English language translation from the original document,

ELASTOMERER FUßBODENBELAG UND VERFAHREN ZU SEINER HERSTELLUNG (ELASTOMER FLOOR COVERING AND METHOD FOR ITS MANUFACTURE)

written in German; and

3. That the attached translation is an accurate English version of such original to the best of his knowledge and belief, AFTER THE CHANGES MENTIONED IN THE ATTACHED ERRATA SHEET ARE MADE.

ROBERT C. FERBER

Subscribed and Sworn to before me this 1st of March, 2005.

Notary Rublic

SHIRLEY O. SAED
Notary Public, State of New York
No. 01SA5072143
Qualified in New York County
Certificate Filed in New York County
Commission Expires January 27



10

15

20

25

### ELASTOMER FLOOR COVERING AND METHOD FOR ITS MANUFACTURE

#### 5 Field of the Invention

The present invention relates to an elastomer floor covering. In particular, it relates to a homogeneous floor covering of cross-linkable, elastomer material, having a directionless pattern. A manufacturing process is also specified.

### Background Information

European Patent A1 0 512 197 describes a floor covering of the species, which is completely free of trapped air. The manufacturing process is characterized in that, using a short screw-type extruding machine including an attached perforated disk, moldings having a size of 1 to 2 mm are continuously produced from two or more varicolored, vulcanizable elastomer materials by knocking off the emerging strands.

These moldings are passed through the gap of a two-roll calender under a constant line pressure, the calender having horizontally arranged rolls capable of being heated and cooled, a homogeneous band being formed, which is vulcanized to form a ready-made floor covering. To keep the line of pressure in the roll nip as narrow as possible, a very low diameter/length ratio of 1:3.5 is preferred.

30 Using this process, floor coverings having a thickness of approximately 2 mm can only be made in a limited width. In the recommended diameter/length ratio of 1:3.5 for the calender, the width of 1250 mm specified in the citation already requires a roll diameter of 370 mm, given a length of

1300 mm. It has been found that, in the manufacture of a 2 mm thick web, such slim rolls bend away from each other in the middle of their axial extent by up to 1 mm and more. This effect intensifies considerably in response to an increasing roll length, or web width, given a constant roll diameter.

Consequently, the resulting web has a thickness of over 2.8 mm in its corresponding central surface area, and only the areas near the edges have the desired calibration thickness of 2 mm.

10

15

20

5

An alternative to this line pressing process is to process the material flat, for example, in a band molding press. European Patent B1 0 046 526 describes such a process, which has the advantage of an exactly equal pressing pressure in all surface areas, and consequently, the possibility of manufacturing webs having a uniform thickness.

In the process, a raw material layer of spread-out thermoplastic particles is continuously compressed flat at 160°C to 220°C and welded together, and is subsequently cooled down and pressed off at an unchanging pressure. In this context, the web width is dependent only on the width of the molding press.

25 If one tries to apply this method - described for thermoplastic materials - to vulcanizable, elastomer materials for floor coverings, webs are produced whose elongation at break, according to DIN 53 504, has values of only 20 to 30%. Such low elongations, however, are inadequate for floor coverings since they must very often be laid on uneven sub-floors and against edge areas, the floor coverings having to adapt to uneven areas and imprecise fits through certain elongation properties so as to avoid destruction.

However, elastomer materials are always to be given preference since, in contrast to thermoplastic materials, they exhibit a resistance to cigarette burns.

5 Summary of the Invention

The object of the present invention is to specify a homogeneous floor covering of cross-linkable, elastomer material, having a multicolored, directionless pattern, which

- 10 in the case of widths of 1 m to 2 m, exhibits only an insignificant change in thickness (maximally 5%) over its entire surface,
  - has a thickness of 1.5 to 3.5 mm,
  - minimum elongation of 60% according to DIN 53 504.

15

25

30

A further object of the present invention is to specify a continuous manufacturing process, making it possible to produce such a floor covering economically.

The property "homogeneous" is defined for floor coverings according to DIN 16 850:

"Homogeneous elastomer coverings, composed of a layer or a plurality of layers of equal composition. They have a multicolored, continuous pattern, or a continuous, uniform color."

This objective is achieved according to the present invention by using a homogeneous floor covering of cross-linkable, elastomer material, having a multicolored, directionless pattern, and having the properties required by the object of the invention, the floor covering, with respect to its total weight, containing 3 to 20 per cent by weight of a copolymer of ethylene including at least one comonomer belonging to the group of

- vinyl esters of saturated carboxylic acids having up to 4
   C-atoms in the acid group,
- unsaturated mono- or dicarboxylic acids,
- esters of unsaturated mono- or dicarboxylic acids having up to 8 C-atoms in the alcohol portion,
- $\alpha$ -olefins having 4 to 10 C-atoms.

5

15

20

25

30

In this context, the ethylene content of the copolymer must be 40 to 95 per cent by weight, the comonomer content 5 to 60 per cent by weight. The melt-flow index of the copolymer must lie between 0.1 and 50.

Copolymer proportions of 3 to 10 per cent by weight turned out to be particularly advantageous, compared to ones having higher copolymer proportions, since then, due to the slight adhesive quality, the mixture can be processed more favorably on the rolls. In addition, the properties of the ready-made floor covering are not substantially affected; copolymer proportions higher than 10 per cent by weight can cause, for example, a worsening of the fire resistance.

The floor covering can be essentially in two embodiments:

In the one case, the granular particles forming it have a single color in and of themselves; in the other case, they have a colored pattern in and of themselves. The visual effect of the surface is a little quieter in the latter variant while the alternative conveys a livelier picture of the surface.

## Embodiment of the Invention

The manufacture of the floor covering according to the present invention is carried out in a generally known manner in that, first of all, granular material particles are produced from

unvulcanized elastomer material, the particles differing from

each other in color, and also being able to have non-homogeneous colors in and of themselves. To this end, corresponding, varicolored rubber mixtures are produced independently of each other, formed into strands using extruding machines, and subsequently granulated into grain sizes of up to 3.0 mm.

Then, analogously to European Patent B1 0 046 526, at least two granular materials differing in color are spread on a band-shaped, horizontally moving carrier. Such carriers can be composed of release paper, steel band, or polytetrafluoroethylene band, and must never adhere to the particles.

The compressing and vulcanization are carried out in a band molding press appropriate for the width of the floor covering to be produced, at 160 to 180°C and at a pressure of 3 to 30 N/mm<sup>2</sup>. Subsequently, the reverse side of the resulting web is polished for the sake of improving the gluing capability.

Essential to the present invention of this process is that, with respect to the total weight of the materials to be processed, 3 to 20 per cent by weight of a copolymer of ethylene including at least one comonomer are mixed with the unvulcanized rubber mixtures prior to their processing.

### Suitable comonomers include

- vinyl esters of saturated carboxylic acids having up to
   4 C-atoms in the acid group,
- 30 unsaturated mono- or dicarboxylic acids
  - esters of unsaturated mono- or dicarboxylic acids having up to 8 C-atoms in the alcohol portion,
  - $\alpha$ -olefins having 4 to 10 C-atoms.

5

20

The ethylene content of the copolymer is 40 to 95 per cent by weight, the rest is comonomer, and the melt-flow index of the copolymer must lie between 0.1 and 50.

- Using copolymer proportions of 3 to 10 per cent by weight reduces the adhesive quality of the material during processing, and improves the fire resistance and mechanical resilience of the finished product.
- 10 With regard to the alternatives of creating the floor covering from granular material particles which have either a single color in and of themselves or a multicolored pattern in and of themselves, corresponding procedures are known to a worker skilled in the art. The making of multicolored patterns is described, for example, in European Patent A1 0 512 197.

  During the extrusion of the material to be reduced to granular particles, care must be taken that the varicolored strips of vulcanized rubber are not subjected to an intermixing of colors.

A suitable device for this is, for example, a conventional short screw-type extruding machine, which is operated at 60 to 100°C. In a specific embodiment, the resulting strands pass through a perforated disk having bores of a diameter of 3 to 5 mm; the strands are knocked off every 1 to 2 mm.

Alternatively, for this purpose, the plasticized mass is permitted to emerge from a slot nozzle of an extrusion head, and the cooled mass is granulated using cutting mills.

The quantity of copolymer contained in the floor covering according to the present invention, i.e., mixed with the elastomer mass prior to processing, is usefully given a lower limit of 3 per cent by weight, since smaller proportions cannot have any essential effect on the extensibility any

more. The upper limit is 20 per cent by weight since higher values would change the Shore hardness of the elastomer material too much; the Shore hardness of the ready-made floor covering should as far as possible be the same as that of the starting mixture.

Using advantageous embodiments, the achievement of the object according to the present invention is explained by the following examples on the basis of comparable measuring results. All examples start from unvulcanized elastomer material composed as follows:

	Sterene-butadiene rubber	100 parts
15	having a sterene content of 23 per cent by weight	
	Sterene-butadiene copolymer	20 parts
	having a sterene content of 85 per cent by weight	
	Precipitated silicic acid	40 parts
20 .	Kaolin	70 parts
	Precipitated chalk	30 parts
	Coumarone-indene resin	15 parts
	Zinc oxide	5 parts
	Stearic acid	1.5 parts
	Sulphuric acid	2.5 parts
25	Polyethylene glycol	3 parts
	Cyclohexyl benzothiazole sulphene amide	2.7 parts
	Tetramethylthiuramdisulfide	0.5 parts
	Pigments	as desired

The particle-size distribution of the granular material is

30	> 2.5 mm	1 %
	> 2.0 mm	33 %
	> 1.4 mm	55 %
	> 1.0 mm	25 %
	> 0.71 mm	6 %

5

The starting mixture has the following parameters:

# Starting mixture

5

as roll compound		as spread layer		
elongation %	Shore hardness	elongation %	Shore hardness	
120	92	30	92	

Table 1

Table 1 reveals how the above characterized elastomer granular material, after the processing in a two-roll calender

10 according to the related art, has an excellent elongation of 120 %, while the same material passing through a band molding press as a spread layer has an elongation of only 30 %, which is inadequate to meet the technical requirements for laying.

15 Table 2 specifies the variants of processing the starting mixture via a band molding press. Here, as in Table 1, the following parameters apply:

	Width of the band molding press	1500	mm
20	Width of the floor covering to be produced	1300	mm
	Pressing pressure	30	$\mathrm{N/mm^2}$
	Pressing temperature	180	°C
	Vulcanization time	4	min

25 As Table 2 clearly reveals, different quantities of a copolymer of ethylene including at least one comonomer (1st column) were added to the mixture. In the following columns of .

Table 2, the comonomer contents are assigned to these copolymer quantities.

It turns out that the copolymers composed and added according to the present invention produce minimum elongations of 60 % according to DIN 53 504 in the floor covering, while the Shore hardnesses remain nearly unchanged with respect to the starting mixture.

E-octene	14 % octene	long.% Shore hardness			92	06
EEA	18 % EA	elong. * Shore hardness elong. * Shore hardness			45 92 40	60 91 60
	50 % VA	elong.% Shore hardness	60 91		160 89 4	9
EVA	28 % VA	elong.% Shore hardness	40 92	40 92	70 91	06 06
18 % VA	addition in elong.% Shore hardness		35 93	1	55 92	7091
Copolymer	addition in	kg	10	15	20	30

Table 2

As Table 2 clearly reveals, different quantities of a copolymer of ethylene including at least one comonomer (1st column) were added to the mixture. In the following columns of Table 2, the comonomer contents are assigned to these copolymer quantities.

It turns out that the copolymers composed and added according to the present invention produce minimum elongations of 60 % according to DIN 53 504 in the floor covering, while the Shore hardnesses remain nearly unchanged with respect to the starting mixture.

When using copolymers including vinyl acetate as comonomer, the rule applies that low vinyl acetate contents require a greater quantity of copolymer to be added to the mixture (over 20 kg, Table 2). Adding these increased quantities, however, does not result in a noticeable reduction of the Shore-A hardness of the product. Nor is the mechanical resilience negatively affected in the process.

20

25

30

5

10

15

However, if the vinyl acetate content in the copolymer is increased to, for example, 28 per cent by weight, the desired tensile strength is already reached at a copolymer addition of 20 kg, as shown in Table 2. In the process, the Shore-A hardness is not disadvantageously reduced.

In the case of even higher vinyl acetate contents (50 per cent by weight), a marked softening of the elastomer mixture can be detected even when small quantities of copolymer (10 kg) are added.

Copolymers behave similarly in conjunction with other comonomers; examples are an ethylene-ethyl acrylate copolymer having an ethyl acrylate content of 18 per cent by weight as

well as an ethylene-octene copolymer having an octene content of 14 per cent by weight. Both copolymers can be obtained on the market. In the case of the comparatively low comonomer proportions, copolymer additions of over 20 kg are necessary to reach the desired minimum elongation. The achieved Shore-A hardnesses, however, lie within the range which can still be tolerated, but at its margin.

#### Patent Claims

1. A homogeneous floor covering of cross-linkable, elastomer material, having a multicolored, directionless pattern, and a thickness of 1.5 to 3.5 mm, characterized in that the floor covering, in the case of widths of 1 m to 2 m, has no change in thickness exceeding + 5% over its entire width, that the floor covering has a minimum elongation of 60% according to DIN 53 504 (tensile test), and that it contains 3 to 20 per cent by weight, with respect to its total weight, of a copolymer of ethylene including at least one comonomer belonging to the group of

- vinyl esters of saturated carboxylic acids having up to 4
   C-atoms in the acid group,
- unsaturated mono- or dicarboxylic acids,
- esters of unsaturated mono- or dicarboxylic acids having up to 8 C-atoms in the alcohol portion, or
- $\alpha$ -olefins having 4 to 10 C-atoms, the ethylene content of the copolymer being 40 to 95 per cent by weight, the comonomer content 5 to 60 per cent by weight, and the melt-flow index of the copolymer lying between 0.1 and 50.
- 2. The floor covering as recited in Claim 1, characterized in that it contains 3 to 10 per cent by weight of a copolymer.
- 3. The floor covering as recited in Claim 1 or 2, characterized in that the granular particles forming it have a single color in and of themselves.
- 4. The floor covering as recited in Claim 1 or 2, characterized in that the granular particles forming it have a colored pattern in and of themselves.

5. A process for manufacturing a homogeneous floor covering of cross-linkable, elastomer material, having a multicolored, directionless pattern, a thickness of 1.5 to 3.5 mm and a width of 1 to 2 m,

comprising the manufacture of granular particles existing in at least two colors which differ from each other, from correspondingly varicolored rubber mixtures produced independently of each other, which are formed into strands using extruding machines, and subsequently granulated into grain sizes of up to 3.0 mm;

further comprising the subsequent spreading of the granular particles on a band-shaped, horizontally moving carrier, the compression and vulcanization in a band molding press appropriate for the width of the floor covering to be produced, at 160 to 180°C and at a pressure of 3 to 10 N/mm², the subsequent polishing of the surface forming the back side of the resulting web,

characterized in that, with respect to the total weight of all materials to be processed, 3 to 20 per cent by weight of a copolymer of ethylene including at least one comonomer are mixed with the unvulcanized rubber mixtures prior to their processing, the comonomer belonging to the group of

- vinyl esters of saturated carboxylic acids having up to 4
   C-atoms in the acid group,
- unsaturated mono- or dicarboxylic acids,
- esters of unsaturated mono- or dicarboxylic acids having up to 8 C-atoms in the alcohol portion, or
- $\alpha$ -olefins having 4 to 10 C-atoms, the ethylene content of the copolymer being 40 to 95% per cent by weight, the comonomer content 5 to 60 per cent by weight, and the melt-flow index of the copolymer lying between 0.1 and 50.
- 6. The process as recited in Claim 5,

characterized in that a copolymer proportion of 3 to 10 per cent by weight is selected.

- 7. The process as recited in Claim 5 or 6, characterized in that granular particles having a single color in and of themselves are produced and used.
- 8. The process as recited in Claim 5 or 6, characterized in that granular particles having a colored pattern in and of themselves are produced and used.

#### Abstract of the Disclosure

A homogeneous floor covering composed of elastomer, varicolored granular particles contains 3 to 20 per cent by weight of a copolymer of ethylene, including at least one comonomer of vinyl esters of saturated carboxylic acids having up to 4 C-atoms in the acid group, including at least one unsaturated mono- or dicarboxylic acid, or including at least one ester of unsaturated mono- or dicarboxylic acids having up to 8 C-atoms in the alcohol portion. In this context, the ethylene content of the copolymer is 40 to 95% per cent by weight, the rest is comonomer, and the melt-flow index of the copolymer must lie between 0.1 and 50. A corresponding manufacturing process is specified.

15

10